STUDIES ON SYNTHESIS, STRUCTURAL, LINEAR AND NONLINEAR OPTICAL PROPERTIES OF PURE AND DOPED ZnO NANOSTRUCTURES

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Introduction

Nanotechnology has been recognized as a revolutionary field comparable to many other advanced fields of science and technology. It is concerned with the behaviour of materials when scaled down to a nanometer (1 to 100 nm). At this nanoscale size, materials begin to exhibit distinct physical and chemical properties compared with those of the bulk materials [1]. Though the last few decades have seen significant breakthroughs in the growth and applications of different kinds of nanomaterials, research is still in progress to meet the growing needs of human society. In this context, semiconductor nanotechnology is being given greater attention as it offers the prospect of significant innovation by providing materials with remarkable properties and functionalities outside the current domain. The reduction of semiconductor dimensions to the nanoscale level improves the device function effectively. Nanostructures, either of metal or metal oxides, have gained immense importance in the era of high-end technology.

Nanomaterials

Nanostructures are the geometrical entities with distinct shape and size with at least one of the dimensions in the nanoscale. They are classified as zero dimensional, one, and 2 dimensional based on their dimensionality. In bulk materials, all the three dimensions have macroscopic scale. If one of the three dimensions is within the range of 1-100 nm then it is called as 2-D nanostructure such as graphene sheets which are 2-D layer of atoms. In 1-D nanomaterials, two out of three dimensions are in the nanoscale regime. It can be nanorods, nanowires, nanotubes. Carbon nanotubes and its composites are one such example of 1-D nano system [2][3][4]. When all the three dimensions are in the nanoscale regime, it is termed as 0-D nanostructures. Nanoparticles have all three dimensions on the nanoscale. It is typically an independent entity which exists in isolation. They can be synthesized with very specific shapes such as spheres, cylinder, cube, flower disks etc. Nanoparticles can also be embedded in a continuous primary phase such as polymer matrix to form a nanocomposite [5].

In the past few decades, semiconductor nanostructures gained significant attention among research groups due to their potential use in wide variety of applications.

Metal oxide nanostructures

Metal oxides are crystalline solids composed of oxygen anions and metal cations. They exist in many different crystal forms. They are classified as semiconductors or insulators based on their electronic band structure. They exhibit unique physico-chemical properties that give rise to many potential applications in different areas of applied physics and chemistry [6]. In this era of nanotechnology, the nanostructures of these oxides have extensively used in many fields of physics, chemistry, materials science, and biotechnology. Primary factors which cause nanoparticles to behave significantly different from conventional bulk materials are increased relative surface area and quantum confinement effect arises when the particle size is less than the Bohr excitonic radius of the corresponding atom [7]. These factors can enhance the properties such as strength, reactivity, electrical and optical characteristics dramatically. Many techniques have been developed to synthesize nanoparticles with controlled size, surface morphology, dimensionality and crystallinity [8].

The method of synthesis has played a crucial role in exploring the novel properties of nanomaterials. The enhanced properties and applications of nanomaterials are possible when nanostructured materials of desired size, shape, composition and crystallinity are made available. Many techniques have been developed to fabricate nanomaterials. In general, growth techniques are classified into two groups; the top-down approach, and the bottom-up approach. The top-down approach involves the breaking down of the bulk material into nanosized structures, for example electron beam lithography, high energy wet ball milling, atomic force manipulation, aerosol spray, etc. The latter refers to building the organic and inorganic nanostructures atom-by-atom and molecule-by-molecule by self-assembly process. Various physical and chemical methods have been developed based on this approach. Pulsed laser deposition, sputtering,

molecular beam epitaxy, thermal vapour deposition are some of the widely used physical methods for the synthesis of nanostructures for industrial applications [9][10][11][12]. The sol-gel synthesis, colloidal precipitation, hydrothermal technique, organometallic chemical route, thermal decomposition etc. are some of the well-known solution phase synthesis reported for the preparation of different kinds of nanoparticles [13][14][15]. However, all the methods have their own drawbacks and involve limitations concerning processing, desired properties, and cost. Therefore, opting for an ideal synthesis method is essential for controlling the properties of the resultant nanostructures.

Zinc oxide

Nanostructures of Zinc Oxide (ZnO) are at the centre of much attention among metal oxides because of their ability to possess distinctive properties. ZnO is an II-VI binary semiconductor compound, crystallizes in stable hexagonal wurtzite structure at ambient conditions. ZnO is a wide bandgap material (3.37 eV) with higher order transmittance in the visible region and it exhibits large excitonic binding energy (60 meV) at room temperature [16]. Also, the processing of ZnO is user-friendly and it is a nontoxic semiconducting material. Moreover, ZnO exhibits non-centrosymmetric structure, thus possessing second and third-order nonlinearities making it a suitable material for optical limiters, mode locking devices, high-speed digital information processing, and electro-optic modulators in the field of nonlinear optics [17]. Apart from this, the surface modification of the ZnO nanoparticles by various biomolecules makes it a suitable candidate for biomedical applications such as drug delivery, magnetic resonance imaging, and biosensors [18]. In addition, ZnO has inherent optical and electrical properties. This makes ZnO a perfect candidate for optoelectronic applications. To improve these properties doping ZnO with external impurities is performed. This ensures the increased stability over different physical and chemical conditions. Also, doping modifies and enhances the optical and electrical properties of ZnO nanoparticles. The doping of external impurities can also result in the formation of defect states in the band structure of the host ZnO. It is also reported that the

doping of external impurities may result in phase segregation [19–22]. Although a lot of work has been done on ZnO nanostructures from both theoretical and experimental perspectives, there are still lot of challenges to be overcome. Thus, it is important to understand in great detail pure and doped ZnO nanostructures in terms of synthesis, properties, and applications.

Specific objectives of the present study

The main objectives of the present study are,

i. To synthesize the ZnO nanostructures with diverse morphologies using hydrothermal technique in the presence of surfactants such as saponin, glutamine, and oleyalamine.
ii. To study the effect of surfactants on the structural, morphological, and optical properties of ZnO nanostructures.
iii. To study the effect of doping of rare-earth ions, Europium (Eu^{3+}) and Erbium (Er^{3+}) on the structural and optical properties of ZnO nanostructures.
iv. To understand the influence of dopant-induced structural defects over the nonlinear optical properties of ZnO by varying the dopant concentrations.
v. To synthesize pure and Fe-doped ZnO nanoparticles by thermal decomposition of a metal-organic precursor under nitrogen atmosphere and to study the effect of synthesis and dopant on the structural, magnetic properties, linear and nonlinear optical properties of the ZnO nanoparticles.

The results of the study are presented and discussed in the thesis. The thesis is broadly divided into seven chapters with several sections in each chapter.

The brief sketch of the thesis is as follows:

Chapter 1: Introduction

The chapter begins with a general introduction to nanomaterials and metal oxide nanostructures. It includes the general properties of ZnO and its utilization in various applications. The theory of nonlinear optics which includes two-photon absorption and intensity-dependent nonlinear refraction has been explained. A brief literature survey on the synthesis of ZnO nanoparticles, surface

modifications by various surfactants, their doped forms, and defects is presented. In the end, specific objectives of the present study are mentioned.

Chapter 2: methods of synthesis and characterization techniques

Chapter 2 deals with the various synthesis and characterization techniques used in the present study. A detailed introduction to the hydrothermal technique and thermal decomposition method is given. A brief note on the structural characterization equipment, namely X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), Transmission Electron Microscopy (TEM), Fourier Transform Infrared microscopy (FTIR) and the optical characterization techniques namely Room Temperature Photoluminescence (RTPL) spectra and UV-VIS spectroscopy. Nonlinear optical properties of synthesized nanoparticles were studied using single beam Z-scan technique and the experimental details are discussed. Also, the effect of dopants on the magnetic properties of the prepared nanoparticles were studied using Vibrating Sample Magnetometer equipment (VSM).

Chapter 3 Surfactant-assisted hydrothermal synthesis of ZnO nanostructures and their structural and optical properties

This chapter describes the role of surfactants in controlling the surface morphology, size distribution of ZnO nanoparticles and to study their structural and optical properties. The morphology of nanoparticles is critical for their efficient use in many applications ranging from biomedicine to sensing and energy. While shape and size are responsible for the properties of ZnO core, the choice of ligands is of utmost importance for the colloidal stability and function of nanoparticles [23]. Stability of the nanoparticles depends on the type of surfactants used during the synthesis. Surfactants are organic molecules that have the ability to adhere to the surface of the nanoparticles and are often used for controlling the size, aggregation, and morphology of the nanoparticles. The nanocrystals with anisotropic shapes such as nanorods, nanowires, nanotubes nanodiscs, branched shapes, etc, can be produced by selecting a suitable

surfactant during the growth process [24]. It is also to be noted that although, different capping agents direct the orientation of particles in different ways, the type of synthesis involved is one of the imperative factors that equally govern the final morphology, microstructure and size of ZnO nanoparticles [25–28]. In the present study, the ZnO nanostructures are synthesized by hydrothermal technique using glutamine, oleylamine and saponin as surfactants. First two belongs to the amine group and saponin is a plant extract. The samples were named as surfactant-free ZnO, OAmZnO, GZnO, and SZnO. The synthesized nanostructures were characterized using XRD, FTIR, FESEM, TEM, UV-visible spectroscopy and RTPL spectroscopy techniques. Nanospheres, hexagonal nanorods, and cylindrical shaped nanorods were formed when grown in the presence of surfactants, saponin, glutamine, and oleylamine respectively (Fig. 1 and Fig. 2). RTPL spectra of GZnO and OAm ZnO showed the presence of DLE emission peaks, whereas these emission peaks were absent in SZnO nanoparticles. These results suggested that the surfactants can effectively be used to tune the structural and optical properties of the ZnO nanostructures.

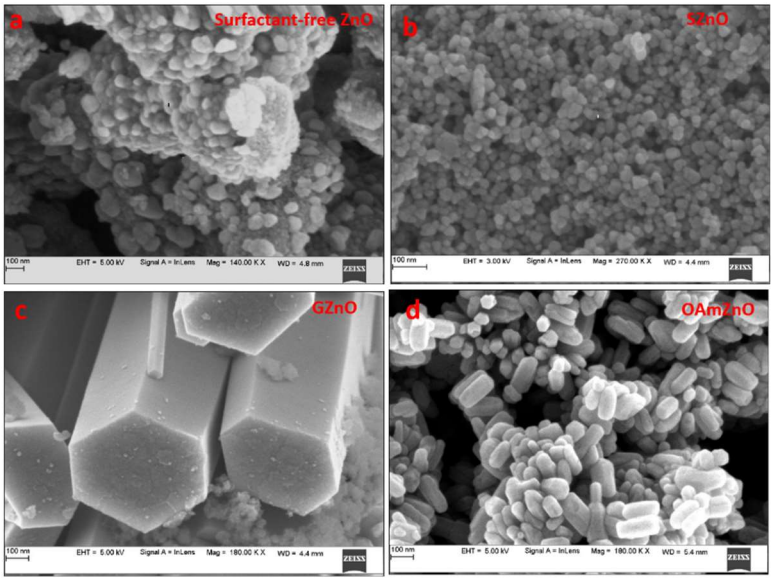

Figure 1. FESEM images of a) Surfactant-free ZnO, b) SZnO, c) GZnO, d) OAmZnO nanostructures

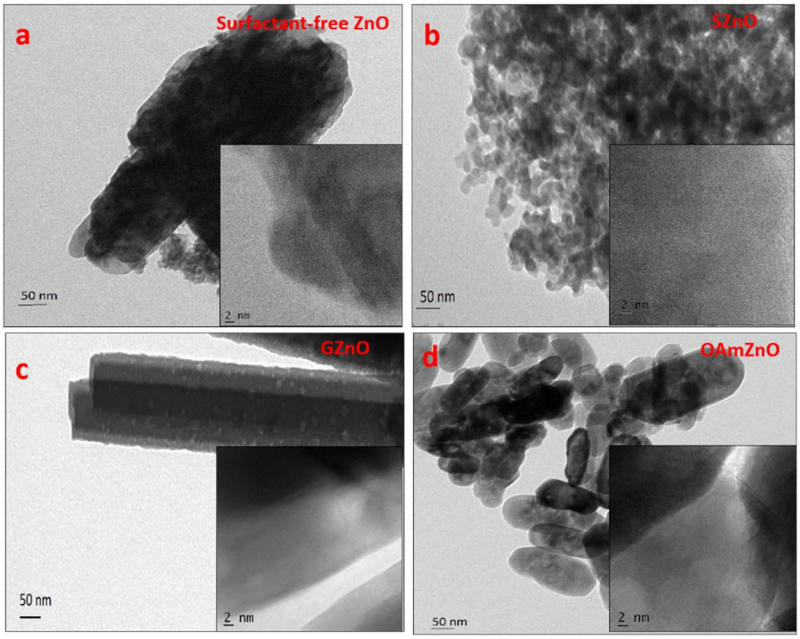

Figure 2. TEM images of a) Surfactant-free ZnO, b) SZnO, c) GZnO, d) OAmZnO nanostructures

Chapter 4: Defects induced nonlinear saturable absorption mechanism in europium doped ZnO NPs synthesized by facile hydrothermal method

In the present chapter, undoped and Eu-doped (1 at.wt%, 3 at.wt% and 5 at.wt%) ZnO NPs were prepared by surfactant-assisted hydrothermal synthesis. The effect of Eu dopant concentration on the structural, linear, and third order nonlinear optical properties of ZnO NPs was studied. The structural and linear optical properties were characterized XRD, FTIR, FESEM, TEM, UV-visible spectroscopy and RTPL spectroscopy techniques. The generation of compressive strain was observed in Eu doped ZnO NPs due to the difference in ionic radii of Zn and Eu. A significant decrease in the energy gap with an increase in the Eu doping concentration was attributed to the formation of localized defect states below the conduction band edge (Fig. 3).

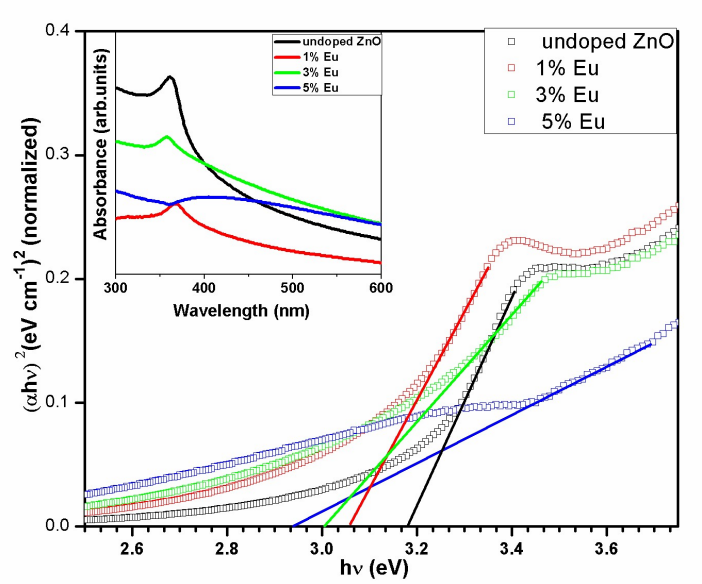

Figure 3. Tauc's plot to determine the energy gap of the undoped and Eu doped ZnO NPs

The third order NLO properties were studied using the single beam Z- scan technique. The Z-scan measurements of undoped ZnO, 1% Eu and 3% Eu doped ZnO NPs exhibited TPA-induced RSA behaviour and self-defocusing mechanism. A strong saturable absorption behaviour is noticed in 5% Eu doped ZnO NPs due to depletion of carriers in the ground state whereas the self-focusing behaviour is due to thermo-optical effects (Fig.4).

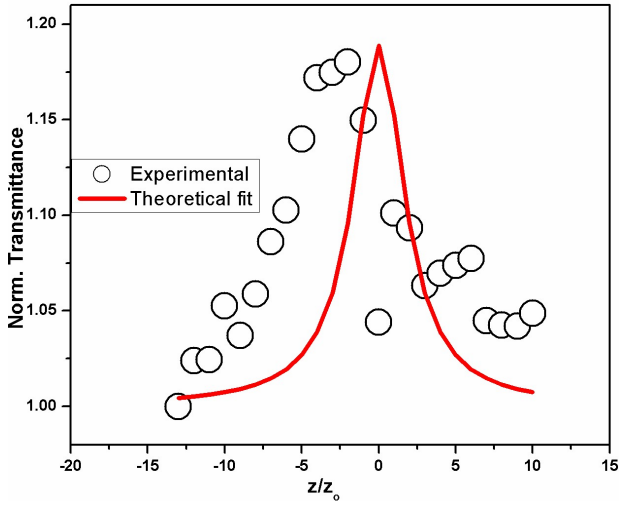

Figure 4. Open aperture Z scan curve of 5% Eu doped ZnO NPs showing the saturable absorption behaviour.

Chapter 5: Phase segregation induced third order nonlinear saturable absorption behaviour in Erbium doped ZnO nanoparticles synthesized by facile hydrothermal method

In the present chapter, undoped and Er doped (1 at.wt%, 3 at.wt% and 5 at.wt%) ZnO nanoparticles were prepared by surfactant-assisted hydrothermal synthesis. The samples were named as PZnO, ZnEr1, ZnEr3, and ZnEr5 for undoped ZnO, 1 at.wt.% Er doped ZnO, 3 at.wt.% Er doped ZnO, and 5 at.wt.% Er doped ZnO nanoparticles respectively. The effect of Eu dopant concentration on the structural, linear, and third order nonlinear optical properties of ZnO nanoparticles was studied. The structural and linear optical properties were characterized XRD, FTIR, FESEM, TEM, UV-visible spectroscopy and RTPL spectroscopy techniques. The third order NLO properties were studied using the single beam Z-scan technique.

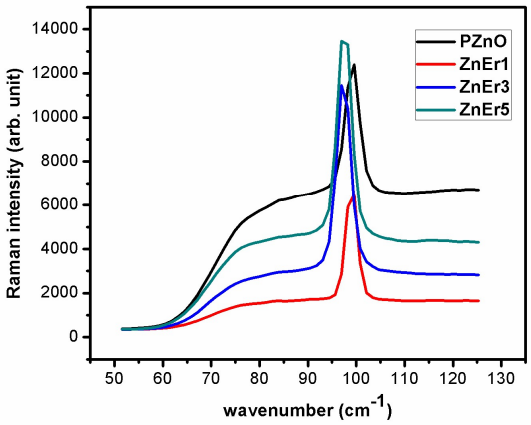

Figure 5. Raman spectra of synthesized nanoparticles showing the variations in intensity and peak position related to E_2 (Low) mode.

The bandgap shrinkage with increasing Er doping concentration is explained based on the formation of extended defect states between the conduction and valence bands in ZnO nanoparticles. The retention of wurtzite structure upon Er doping is confirmed by the presence of E_2^{low} and E_2^{high} Raman modes in all the nanoparticles. The disappearance of A_1(TO) mode in ZnEr3 and ZnEr5 nanoparticles shows the deteriorated polar lattice bond strength (Fig. 5). The

phase segregation observed from the XRD spectrum of ZnEr5 nanoparticles is further confirmed by the detection of local vibrational mode (LVM). The dominance of surface defects and native defects in Er doped ZnO nanoparticles is revealed by the PL spectra. Further, the strength of the NBE emission decreases with increasing Er doping concentration. The open and closed aperture Z scan curves of ZnEr5 nanoparticles switch from RSA to SA and self-defocusing to self-focusing behaviours respectively (Fig. 6). The present study sheds the light on impact of lanthanide doping on the optoelectronic properties of ZnO nanoparticles.

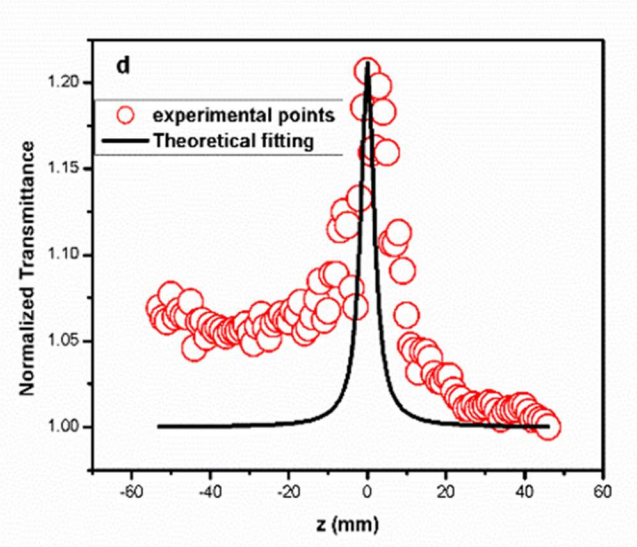

Figure 6. Open aperture Z- scan curve of ZnEr5 showing the saturable absorption behaviour

Chapter 6: Surface dangling bonds foisted atypical deep ultraviolet emission in Fe doped ZnO nanoparticles synthesized by thermal decomposition method under nitrogen atmosphere

In this chapter, the suitability of ZnO nanoparticles in the spintronics applications is tested by incorporating Fe and thermal decomposition technique as dopant and synthesis method respectively. Fe, being one of the transition metals has become a promising candidate to uplift the magnetic behaviour of the ZnO nanostructures [29]. Further, the studies have shown that, the intrinsic defects in ZnO originating during synthesis are responsible for the magnetic

behaviour. The intrinsic magnetism in ZnO is attributed to oxygen vacancies and zinc interstitials [30]. The thermal decomposition method can be effectively used to synthesis monodispersed metal oxide nanoparticles with great controllability over particle sizes and morphology of the nanoparticles. The control over the particle sizes can be effectively used to tune the electronic structure of the nanoparticles. Therefore, thermal decomposition technique is one of the finest nanoparticles synthesis methods to achieve greater control over the particle size and particle size related parameters such as morphology, quantum confinement effect, energy gap values, and electronic structure.

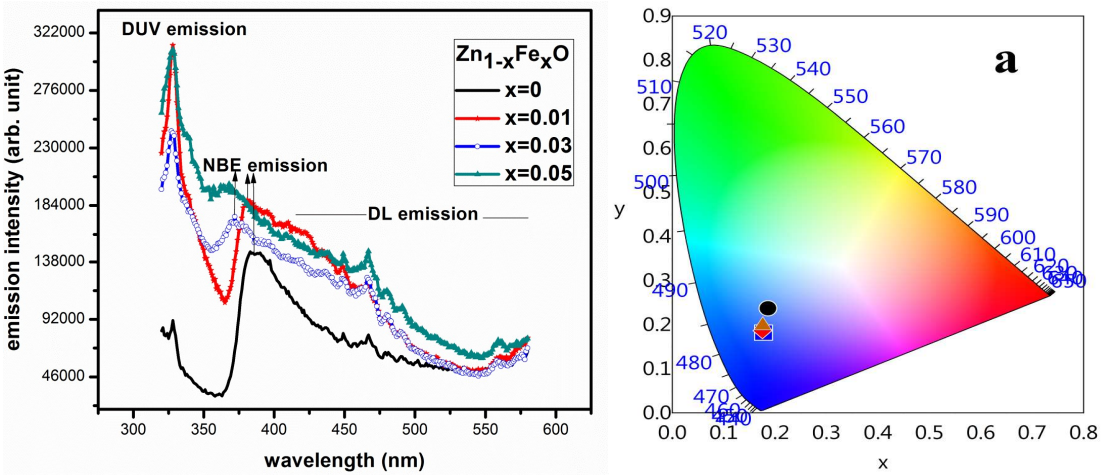

Figure 7. Room temperature emission spectra of $Zn_{1-x}Fe_xO$ nanoparticles revealing deep ultraviolet emission and 1931 CIE diagram of $Zn_{1-x}Fe_xO$ nanoparticles showing the dominant blue emission

The present work also sheds light on the unusual Deep Ultraviolet (DUV) emission noticed in undoped and Fe doped ZnO nanoparticles. The role of thermal decomposition synthesis technique and its influence over the electronic structure of nanoparticles is discussed. The DUV emissive materials are having great importance in several areas, such as radiation hard UV sources, UV therapy, UV sterilization, UV water treatment etc. The DUV emissive semiconductors such as GaN, InGaN, and AlGaN are developed primarily for high density optical storage. Therefore, a broad range of DUV emission applications provide a huge scope to develop new UV emissive semiconducting materials. The wide band gap nature and large exciton binding energy (60 meV)

associated with ZnO can be used to produce DUV emission. However, this requires large band gap modulation. These studies open the possible use of ZnO based nanostructures in the UV emitting studies.

In this Chapter, the $Zn_{1-x}Fe_xO$ (x=0, 0.01, 0.03, 0.5) nanoparticles with different Fe doping concentrations are synthesized using the thermal decomposition technique under an inert nitrogen atmosphere. The $Zn_{1-x}Fe_xO$ nanoparticles showed rarely observed deep ultraviolet emission due to the formation of O 2p related states around zinc vacancies at the surface (Fig. 7). The magnetization properties of the synthesized nanoparticles are studied at room temperature. The M-H curves of all the samples show the coexistence of the ferromagnetic and paramagnetic nature. The colour parameters such as colour coordinates (x, y), dominant wavelength, and colour purity of the samples are derived from the chromaticity diagram. The dominant wavelengths of all the samples fall in the blue region. Further, the presence of surface states in the band gap region of $Zn_{1-x}Fe_xO$ nanoparticles is confirmed by nonlinear Z- scan measurements.

Chapter 7: Conclusions and scope for the future work:

A comprehensive conclusion of the entire investigations carried out in the present study and the future scope is presented in this chapter.

Major conclusions drawn by the systematic studies on the synthesis, properties and applications of undoped ZnO, doped ZnO nanoparticles are the following:

- The ZnO nanostructures were synthesized by employing a hydrothermal technique using saponin, L-glutamine, and oleyl amine as surfactants. The effect of surfactants on the morphology and optical properties of ZnO nanostructures has been studied. The shape of the nanostructure changes from spherical to hexagonal nanorods and then cylindrical nanorods, respectively, when surfactants change saponin to L-glutamine and then oleylamine. The optical band gap values of the ZnO nanostructures are varied between 3 to 3.6 eV. Further, the different nanostructures exhibit different emission properties due to the presence of constituent molecules of surfactants on the surface of ZnO nanostructures. The ZnO nanoparticles were also synthesized by the thermal decomposition

technique under an inert nitrogen atmosphere. High purity, highly crystalline nanodisks were obtained using this method. The ZnO nanodisks showed rarely observed deep ultraviolet emission due to the formation of O2p states around the zinc vacancies at the surface. The surfactants, as well as the method adopted for the synthesis, affect the properties of nanostructures significantly, and thus the optimization of the synthesis parameters is very important for the desired applications.

- The rare earth ions, Eu^{3+} and Er^{3+} doped ZnO nanostructures with 1 at wt %, 2 at wt %, and 5 at wt % doping ratios were prepared using the surfactant-assisted hydrothermal technique. It was observed that the incorporation of dopants into the ZnO host matrix brought changes in the structural, linear and nonlinear optical characteristics significantly. The difference in ionic radii between the dopants (Eu^{3+} and Er^{3+}) and the host Zn^{2+} ionic sites resulted in the compressive strain in the ZnO lattice and contributed to the enhancement of the defect states. Further, the phase segregation observed from the XRD spectrum of ZnEr5 nanoparticles is confirmed by the detection of local vibrational mode (LVM) in the Raman spectra. The bandgap shrinkage with the increasing Eu^{3+} and Er^{3+} doping concentration is explained based on the formation of extended defect states between the conduction and valence bands in ZnO nanoparticles. The enhancement of defect level emission (DLE) peaks in room temperature photoluminescence (RTPL) spectra of 5% Eu^{3+} and 5% Er^{3+}-doped ZnO samples confirmed the defect-assisted electronic transitions.
- The nonlinear optical properties of the undoped and lanthanides (Eu, Er) doped ZnO nanoparticles were studied using single beam Z-scan technique using continuous wavelength laser. The study showed interesting results with respect to nonlinear absorption constant and nonlinear refraction in the nanoparticles. The open aperture Z-scan measurements showed Two photon absorption (TPA) induced reverse saturable absorption (RSA) behaviour in undoped, 1% and 3% Eu and Er-doped ZnO nanoparticles Further, strong saturable absorption (SA)

behaviour was observed in 5% of Eu and Er doped ZnO nanoparticles due to the depletion of charge carriers as a result of higher concentrations of defect states. Further, strong saturable absorption (SA) behaviour was noticed in 5% Eu and Er doped ZnO nanoparticles as a result of the depletion of charge carriers in the ground state due to the higher concentrations of defect states. Further, the Z-scan measurements of undoped, 1%, and 3% Eu and Er doped ZnO nanoparticles showed a self-defocusing mechanism and 5% Eu and Er doped samples showed a self-focusing nonlinearity. The observed self-focusing mechanism is primarily attributed to the thermal lensing effect caused by enhanced defect concentration. Moreover, Er-doped ZnO nanoparticles showed higher order of nonlinear susceptibilities (10^{-4} esu) compared to Eu-doped ZnO nanoparticles (10^{-7} esu). The present study sheds light on the impact of rare earth ions doping on the optoelectronic properties of ZnO nanoparticles.

- The $Zn_{1-x}Fe_xO$ nanoparticles with different Fe doping concentrations are synthesized using the thermal decomposition technique under an inert nitrogen atmosphere. SEM images showed the stacked plate-like morphology upon Fe inclusion. The $Zn_{1-x}Fe_xO$ nanoparticles showed the deep ultraviolet emission due to the formation of O 2p related states around zinc vacancies at the surface. The crystallite size was found to increase with increasing Fe concentration. The smaller energy gap values in Fe doped samples reveal the dominance of surface states. The coexistence of the ferromagnetic and paramagnetic nature in all the samples suggests the samples could be used as dilute magnetic semiconductors in spintronics applications. The enhancement in the nonlinear parameters upon Fe inclusion ascertained the possible applications in the optical power limiting devices. Further, the colour parameters derived from the 1931 CIE diagram suggest that $Zn_{1-x}Fe_xO$ nanoparticles could be used as blue phosphors in LED applications.

Future scope:

The work presented in this thesis can be extended in several directions and is briefly discussed here.

- The study can be extended to doping ZnO with other rare earth ions at different dopant concentrations and analyze their effect on the structural, optical, electrical, and nonlinear properties.
- The saturable absorption behaviour in doped ZnO nanoparticles may find applications in laser cavities for passive Q-switching.
- The rare-earth ions show high luminescence, they can be used as co-dopants with Fe for ZnO nanoparticles and they may find applications in magnetic resonance imaging.
- Water contamination due to organic pollutants is a major cause of health problems, hence the study can be extended to find applications in the photodegradation of organic dyes.
- The gas sensing properties of the nanostructures can be studied because doped ZnO nanostructures are used in various gas sensing applications. The various other synthesis techniques can also be considered to improve the sensing efficiency of the nanostructures.

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In Research Journals/conference proceedings

1. **Vinoditha. U**., Sarojini. B. K., Sandeep. K. M., Narayana, B., Maidur. S. R., Patil. P. S., & Balakrishna K. M. "Defects-induced nonlinear saturable absorption mechanism in europium-doped ZnO nanoparticles synthesized by facile hydrothermal method" *Applied Physics A* (**2019**), *125*(6), 1-11.

2. **Vinoditha. U.,** Sarojini. B. K., Sandeep. K. M., Narayana. B., & Balakrishna. K. M. "Phase segregation induced third order nonlinear saturable absorption behaviour in Erbium doped ZnO nanoparticles synthesized by facile hydrothermal method" Physica *E: Low-dimensional Systems and Nanostructures* (**2020**), 124, 114281.

3. **Vinoditha. U.,** Sandeep. K. M., Sarojini. B. K., Narayana. B., & Balakrishna. K. M. "Surface dangling bonds foisted atypical deep ultraviolet emission in $Zn_{1-x}Fe_xO$ nanoparticles synthesized by thermal decomposition method under nitrogen atmosphere" *Journal of Alloys and Compounds* (**2021**), 867, 158951.

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